ELECTRODE MOUNTING FOR

POTENTIOSTATIC ANODIC POLARIZATION STUDIES

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Electrodes, especially soft and hard metals and thin sheet specimens, are often partially encased in resins, waxes, or varnishes to simplify mounting.[1-6] Although it has been recognized that chemical contamination may occur with such procedures,[7] we have observed that it is also important to avoid crevices at the electrode-resin interface during potentiostatic anodic polarization measurements. The presence of crevices introduces pronounced errors in measured passive current densities.

Samples of an arc melted, 10Cr- 10Ni- Fe alloy were mounted with the Stern-Makrides compression gasket assembly.[8] Some electrodes were mounted directly, others were partially encased or coated with various resins prior to mounting. Exposed surfaces were ground on 2/0 emery paper, rinsed in distilled water, and immersed in the test solution for one hour prior to polarization measurements. Potentiostatic anodic polarization measurements were performed in hydrogen-saturated, normal sulfuric acid at 25°C, using an identical potential step technique[9] for each specimen. Current was measured after 5 minutes at each potential.

Results of the above experiments are shown in Figure 1. Polarization behavior in the active region is unaffected by mounting material. However, all resins except heat-cured epoxy

and alkyd types, markedly increase apparent passive current density. This is due to the presence of fine crevices at the electrode-resin interfaces as shown in Figure 2. These crevices, probably caused by resin shrinkage, enlarge during anodic polarization due to selective dissolution.

In several experiments, auxiliary Luggin probes were sealed into the resin-mounted specimens at the base of the electrode-resin crevice. The potential at these areas remains in the active region (less than -0.300 volt vs. S.C.E.), although the remainder of the specimen is maintained at very noble potentials (up to 1.60 volt vs. S.C.E.). This large potential gradient is caused by the narrow electrolyte path. Although the area within crevices may be small, current density is high, and the effect on measured passive currents is pronounced.

The above results indicate that crevices in, or adjacent to, electrodes must be avoided during potentiostatic anodic polarization measurements. Specimens should be mounted with the Stern-Makrides compression gasket, in heat-cured epoxy resin, in alkyd varnish or other methods which ensure freedom from microscopic crevices.

ACKNOWLEDGMENT

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LIST OF FIGURES

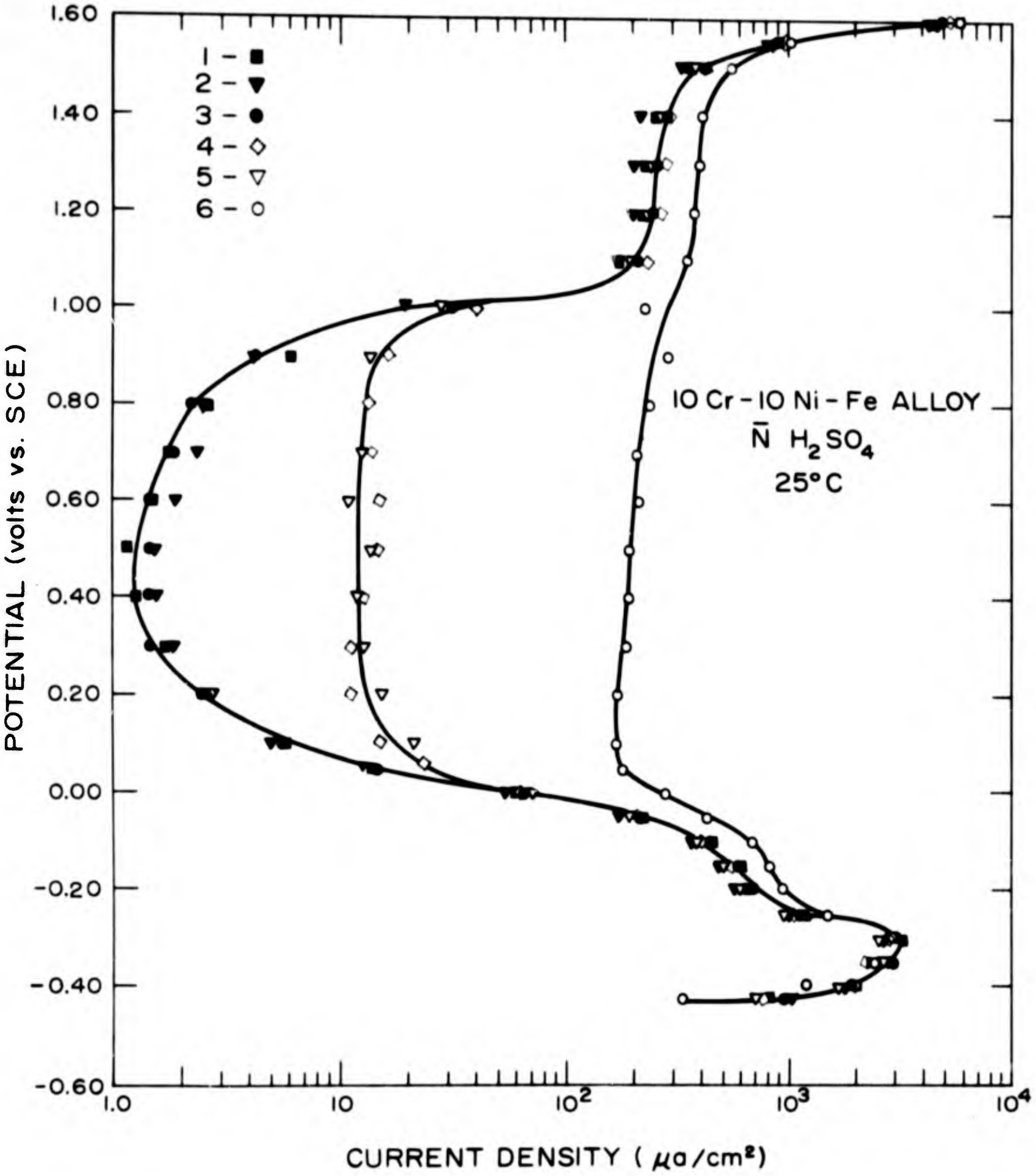
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0.00
-0.20
-0.40
-0.60
1 - ■
2 - ▼
3 - ●
4 - ◇
5 - ▽
6 - ○
10 Cr - 10 Ni - Fe ALLOY
N̄ H2SO4
25°C
POTENTIAL (volts vs. SCE)
1.0
10
10²
10³
10⁴
CURRENT DENSITY (μa/cm²)

FIG.1

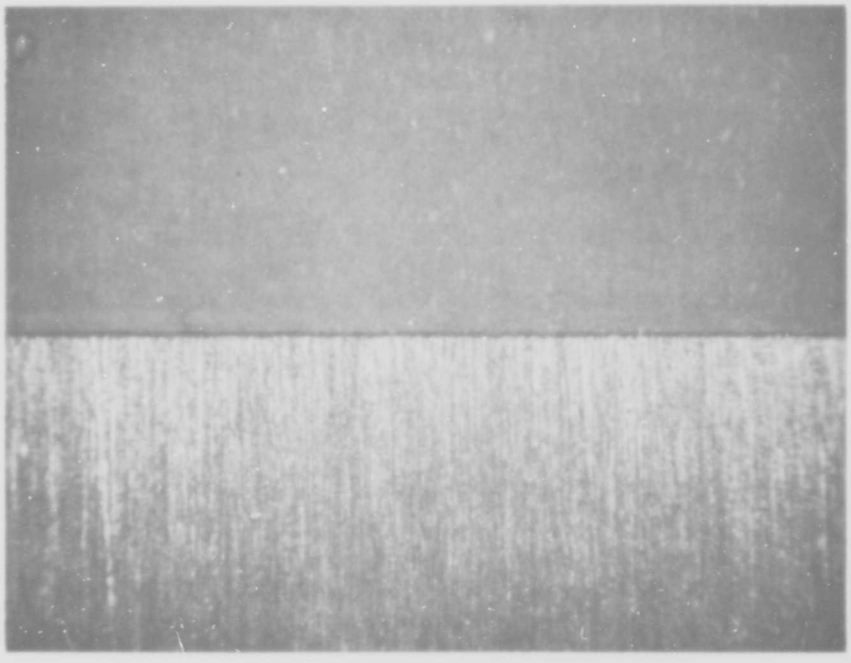

FIGURE 2